

EQUIPMENT

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FACILITY FOR MAKING QUARTZ GLASSES IN VACUUM AND REACTIVE-GAS MEDIUM

S. A. Popov,¹ R. Sh. Nasyrov,^{1,2} and A. S. Lebedev¹

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The Granat-2M growth facility has been updated for synthesizing different quartz glasses by the “gas – vacuum in vacuum” scheme. Its prototype is a commercial facility used for floating ultrapure monomineral and doped quartz glasses by the KS-4V glass production technology.

Key words: quartz glass, ultrapure, monomineral, doping, melting furnace, ampoule, atmosphere, chemical purification, updating, evacuation system.

Advancement of the leading fields in science and technology — optics and illumination engineering, aviation and astronautics, high-purity materials chemistry and instrument building, fiber-optic communications technologies, nuclear power, medicine, and so forth — is determined largely by the usage of fused quartz materials, including glasses.

This is stimulating large-scale scientific and technological research on the possibilities of enriching natural quartz and using it to make ultrapure monomineral and doped quartz glass by unconventional float methods. For purposes of such research a melting furnace for floating different quartz glasses using the “gas – vacuum in vacuum” scheme was developed by updating the well-known Granat-2M growth facility (Fig. 1). Its prototype is a commercial facility used for floating ultrapure (UP) monomineral and doped quartz glasses by KS-4V glass production technology developed at the Institute of the Chemistry of Silicates of the Russian Academy of Sciences.

The starting material — synthetic silicon dioxide (SSD) or quartz crumb — is poured into a vertically positioned quartz glass ampoule. The ampoule is a cylindrical retort to which stems are welded at the top and bottom (Fig. 2). The top stem is used for loading powdered starting material into the ampoule and the bottom stem is used for evacuating the ampoule. A filter, sintered from coarse-grain quartz glass crumb and sealed into the bottom evacuation stem, prevents

the starting material from pouring out of the retort. The top (feeding) stem is sealed off after the ampoule is filled with starting material, and the ampoule is placed into the melting chamber of the furnace. The evacuation stem at the exit from the furnace is sealed with a lens-shaped seal and connected to the ampoule’s high-evacuation and gas admission system.

To prevent the melt from cracking, the ampoule is placed inside a protective cylinder (which is also shape-forming) made of 0.1 mm thick molybdenum metal sheet.

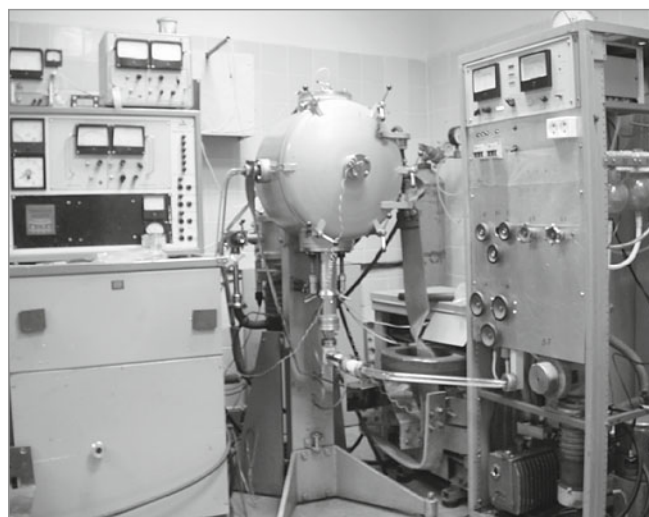


Fig. 1. Granat-2M facility.

¹ Institute of Mineralogy, Ural Branch of the Russian Academy of Sciences, Miass, Chelyabinsk Oblast, Russia.

² E-mail: roudolf@ilmeny.ac.ru.

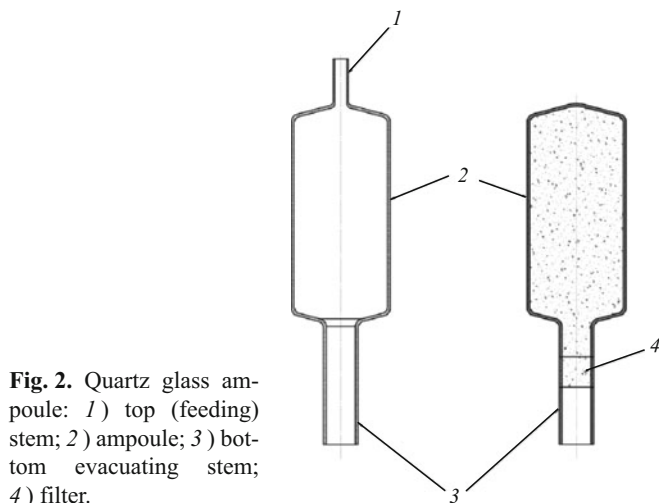


Fig. 2. Quartz glass ampoule: 1) top (feeding) stem; 2) ampoule; 3) bottom evacuating stem; 4) filter.

To prevent the ampoule from rupturing when the chamber is evacuated, the pressure in the chamber is maintained 10–20 kPa higher than the pressure in the ampoule.

As a result of updating the facility, the volume of the high-temperature zone of the Granat-2M melting chamber was increased, which made it possible to use a 70 mm in diameter, 200 mm high ampoule for melting glass.

Figure 3 displays the system for evacuating the chamber and feeding inert gases into it. It consists of a forevacuum 1 and oil-diffusion 2 pumps, a high-vacuum seal 3, and a three-way switch 4. A double-throw switch 5 with a compound pressure and vacuum gauge 6 and two pressure transducers 7 is installed to measure the vacuum or pressure inside the chamber. Inert gas is admitted into the chamber through a special valve 8. A separate system for high-vacuum evacuation and gas admission was assembled to evacuate the ampoule and, if necessary, admit inert or active gases into it. It includes forevacuum and high-vacuum evacuation and gas admission blocks.

The forevacuum block for evacuating the ampoule consists of a NVR-5DM forevacuum pump 9, a nitrogen trap 10, and valves 11, 12; its function is to pre-evacuate the ampoule to residual pressure 10 kPa. The nitrogen trap is installed to keep oil vapors from the forevacuum pump out of the ampoule and to freeze-out corrosive gases evacuated from the ampoule, for example, chlorine, when such gases are used in the melting process.

The high-vacuum ampoule evacuation block contains the following: an NVR-5DM forevacuum pump 9, a fore tank 13, an NVD-100 oil-diffusion pump 14, a slide gate 15, a nitrogen trap 16, and valves 17, 18. The pressure is measured with a VIT-3 vacuum gauge and PMT-2 and PMI-2 pressure transducers 19.

Gases are admitted into the ampoule from a gas-filled tank, through a gas regulator, nitrogen trap 20, valve 21, and membrane filter 22. The pressure is controlled with a combination pressure and vacuum gauge 23. The membrane filter with 0.2 μm pores removes dust-like inclusions from the

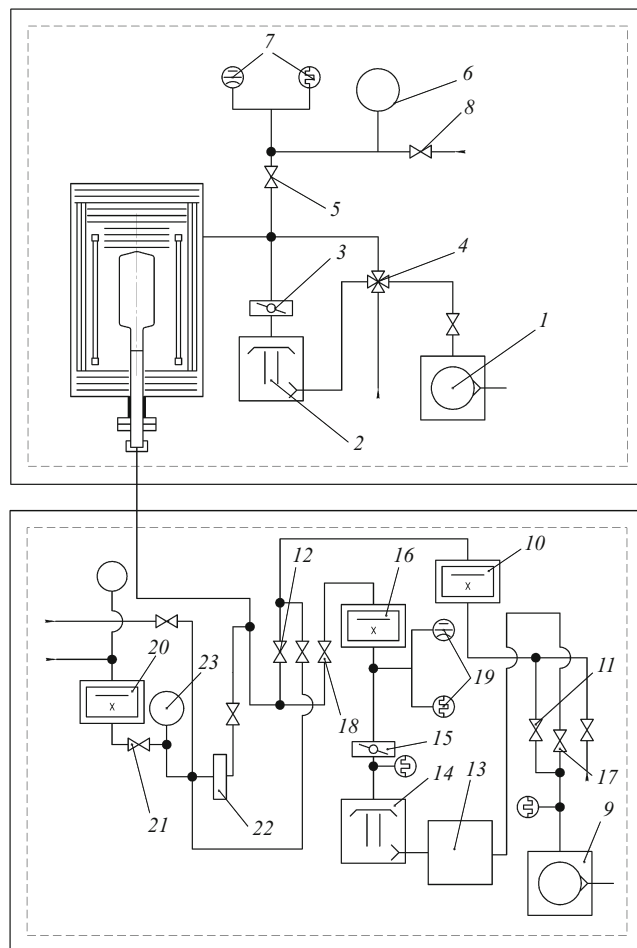


Fig. 3. System for evacuating the chamber and feeding inert gases into it.

gases. The nitrogen trap 20 is intended to dry (by freezing-out) gases admitted into the ampoule at temperature -50°C .

The starting material is doped with different, including rare-earth, elements by introducing the base alloy at the stage where a polysilicic acid (PSA) sol is obtained, followed by processing the sol by the technology used to prepare the starting materials for making KS-4V quartz glass, which makes it possible to make glass with maximum composition homogeneity. One-component and doped quartz glasses are floated in vacuum or in an atmosphere with a prescribed composition in order to chemically purify the starting material by means of reactive gases or to retain the base alloy element in the glass matrix during melting, which, naturally, is impossible during melting in the vacuum medium of the melting chamber of the furnace.

Floats of ultrapure quartz glass, doped with samarium, phosphorus, and neodymium oxides with aluminum were successfully accomplished in the facility. Work on obtaining ultrapure quartz glass doped with rare-earth elements as well as boron and phosphorus for producing special purpose light guides is now in progress.